Designation: D8491 − 22

# Standard Test Method for Recovered Carbon Black—Rheological Non-Linearity of a Rubber Compound by Fourier Transform Rheology[1]

This standard is issued under the fixed designation D8491; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method provides a measure of rheological non-linearity of a rubber compound filled with rCB to assess its reinforcement capabilities. This test method requires the use of a sealed cavity rotorless oscillating shear rheometer for the measurement of the torque with increasing sinusoidal strain applied to an uncured rubber compound containing significant amounts of colloidal fillers, such as recovered carbon black, alone or as blend with virgin carbon black.

1.2 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1485 Practice for Rubber from Natural Sources—Sampling and Sample Preparation

D3191 Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures

D3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)

D3896 Practice for Rubber From Synthetic Sources—Sampling

D6204 Test Method for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

D8059 Test Method for Rubber Compounds—Measurement of Unvulcanized Dynamic Strain Softening (Payne Effect) Using Sealed Cavity Rotorless Shear Rheometers

## 3. Significance and Use

3.1 This test method provides a measure of rheological non-linearity for filled rubber compounds under oscillatory shear conditions: the normalized 3rd harmonic of the torque $I_{3/1}$.

3.2 Rheological linearity means that the modulus is a function of frequency only. The shear modulus dependency on both frequency and amplitude of a dynamic deformation is a non-linear, rheological effect. Filled rubbers show a strain dependency of the modulus known as Payne effect. A test method for evaluating the Payne effect can be found in Test Method D8059.

3.3 One of the main contributions to the Payne effect is the so-called polymer-filler interaction in the range of mid amplitude oscillation shear, MAOS. The MAOS amplitude range is defined as the range where $I_{3/1}$ is already measurable and increases according to a scaling law, for example, $I_{3/1} \sim \gamma^2$. It has been shown that FT rheological measurements are very sensitive to changes in this interaction, and that it is possible to quantify the influence of the filler type and content on the nonlinearity. Interactions between the polymer and particle surface and between the filler particles create a network structure in the compound that not only increases the elasticity of the system but also introduces nonlinear contributions to the stress response.[3]

## 4. Apparatus

4.1 Torsion Strain Rotorless Oscillating Rheometer with a Sealed Cavity in accordance with Test Method D6204, operated in the test mode called strain sweep, in which the strain



[1] This test method is under the jurisdiction of ASTM Committee D36 on Recovered Carbon Black (rCB) and is the direct responsibility of Subcommittee D36.70 on rCB Testing in Rubber.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Schwab, L. et al., "Fourier-Transform Rheology of Unvulcanized, Carbon Black Filled Styrene Butadiene Rubber," *Macromol. Mater. Eng.*, 2016, 301, pp. 457–468.

amplitude is programmed to change in steps under constant frequency and temperature conditions.

4.2 Ensure that the oscillating rheometer is capable of performing a Fourier Transform with the appropriate sampling rate and torque sensitivity.

4.3 Check the input signal before performing this test. The sinusoidal strain signal must be free of higher harmonics. Specifically, the 3rd harmonic of the strain at $\gamma = 32$ % must be maximal 0.25 % of its 1st harmonic (excitation frequency) when testing a reference test specimen, like an unfilled butyl rubber. If this is not the case, request a calibration of the rheometer by the manufacturer.

## 5. Sampling

5.1 A reference compound for evaluation of carbon black in SBR in accordance with Test Methods D3191 shall be used in this test method. For this purpose, substitute the virgin carbon black in the formula by recovered carbon black or by the corresponding filler blend. Alternatively, the reference compound for carbon black evaluation in natural rubber (NR) according to Test Methods D3192 might be used, bearing in mind that the natural variations of NR can increase the variability of the test results.

5.2 The sample shall be taken from the reference compound as required by the mixing method or other sampling instructions. Condition a raw rubber sample in accordance with Practice D1485 or Practice D3896 until it has reached room temperature (23 ± 3°C (73 ± 5°F)) throughout.

5.3 The test specimen shall be circular with a mass of about 5 to 6 g corresponding to a volume of 4 to 6 $cm^3$, see Test Method D6204.

## 6. Procedure

6.1 Place the test specimen in the rheometer chamber and close the chamber. Closing pressure: 450 kPa (4.5 bar).

6.2 Perform a pre-conditioning step with the following parameters:
6.2.1 Temperature: 80.00, in °C.
6.2.2 Offset: 0.00 %.
6.2.3 Pressure: 450 kPa (4.5 bar).
6.2.4 Time: 4.00 min.

6.3 Perform a strain sweep with the following parameters:
6.3.1 Frequency: 0.20 Hz.
6.3.2 Temperature: 80.00, in °C.
6.3.3 Offset: 0.00 %.
6.3.4 Pressure: 450 kPa (4.5 bar).
6.3.5 Pre-cycles: 3.
6.3.6 Amplitude steps $\gamma$, in %: 8, 16, 32, 64, 128.

## 7. Calculations

7.1 Calculate the non-linearity parameter $I_{3/1}$ as ratio of the absolute value of the 3rd harmonic of the torque $S_3$ and the absolute value of the complex torque S* (1st harmonic) at a frequency $\omega$ and a shear strain $\gamma$:

$$I_{3/1}(\gamma) = \frac{|I(3\omega,\gamma)|}{I^*(\omega,\gamma)} = \frac{|S_3(\gamma)|}{|S^*(\gamma)|} \quad (1)$$

7.2 Express the value of $I_{3/1}$ in % by multiplying the ratio with the factor 100.

## 8. Report

8.1 Report the following information:
8.1.1 A full description of the sample or test specimen(s), or both, including their origin.
8.1.2 Type and model of oscillating rheometer.
8.1.3 The temperature and time for the pre-conditioning step.
8.1.4 The frequency in Hertz and temperature in degrees Celsius for the strain sweeps.
8.1.5 Report the non-linearity parameter $I_{3/1}$ in % for $\gamma = 32$ %.

## 9. Precision and Bias

9.1 A precision and bias statement has not been completed for this test method at this time.

## 10. Keywords

10.1 FT rheology; rCB; reinforcement; RPA